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3 **Method Name: Quantitation of Curcuminoids**

4
5 **Intended Use:** Reference method for cGMP compliance.

6
7 **1. Purpose:** AOAC SMPRs describe the minimum recommended performance characteristics to be
8 used during the evaluation of a method. The evaluation may be an on-site verification, a single-
9 laboratory validation, or a multi-site collaborative study. SMPRs are written and adopted by AOAC
10 Stakeholder Panels composed of representatives from the industry, regulatory organizations,
11 contract laboratories, test kit manufacturers, and academic institutions. AOAC SMPRs are used by
12 AOAC Expert Review Panels in their evaluation of validation study data for method being considered
13 for *Performance Tested Methods* or *AOAC Official Methods of Analysis*, and can be used as
14 acceptance criteria for verification at user laboratories.

15
16 **2. Applicability:**

17 The method will be able to separate and quantify each individual curcuminoid, (curcumin,
18 demethoxycurcumin, and bis-demethoxycurcumin) in turmeric [*Curcuma longa* Linn.] dietary
19 ingredients and dietary supplement finished products containing turmeric, alone or in combination
20 with other dietary ingredients.

21
22 **3. Analytical Technique:**

23 Any analytical technique(s) that measures the analytes of interest and meets the following method
24 performance requirements is/are acceptable.

25
26 **4. Definitions:**

27
28 **Analytes**

29 **Curcumin**

30 IUPAC name: (1E,6E)-1,7-Bis(4-hydroxy-3-methoxyphenyl)-1,6-heptadiene-3,5-dione. CAS
31 registry number: 458-37-7. See figure 1 for molecular structure.

32
33 **Demethoxycurcumin**

34 IUPAC name: (1E,6E)-1-(4-Hydroxy-3-methoxyphenyl)-7-(4-hydroxyphenyl)hepta-1,6-diene-3,5-
35 dione. CAS registry number: 24939-17-1. See figure 2 for the molecular structure of demethoxy-
36 curcumin.

37
38 **Bisdemethoxy-curcumin**

39 IUPAC name: (1E,6E)-1,7-Bis(4-hydroxyphenyl)hepta-1,6-diene-3,5-dione. CAS registry number:
40 24939-16-0. See figure 3 for molecular structure.

41
42 **Dietary Ingredients**

43 A vitamin; a mineral; an herb or other botanical; an amino acid; a dietary substance for use by man
44 to supplement the diet by increasing total dietary intake; or a concentrate, metabolite, constituent,
45 extract, or combination of any of the above dietary ingredients.¹

46

¹ Federal Food Drug and Cosmetic Act §201(ff) [U.S.C. 321 (ff)]

47 **Dietary supplements**
48 A product intended for ingestion that contains a "dietary ingredient" intended to add further
49 nutritional value to (supplement) the diet. Dietary supplements may be found in many forms such as
50 tablets, capsules, softgels, gencaps, liquids, or powders.

51
52 **Limit of Quantitation (LOQ)**
53 The minimum concentration or mass of analyte in a given matrix that can be reported as a
54 quantitative result.

55
56 **Quantitative method**
57 Method of analysis which response is the amount of the analyte measured either directly
58 (enumeration in a mass or a volume), or indirectly (color, absorbance, impedance, etc.) in a certain
59 amount of sample.

60
61 **Repeatability**
62 Variation arising when all efforts are made to keep conditions constant by using the same
63 instrument and operator and repeating during a short time period. Expressed as the repeatability
64 standard deviation (SD_r); or % repeatability relative standard deviation (%RSD_r).

65
66 **Reproducibility**
67 The standard deviation or relative standard deviation calculated from among-laboratory data.
68 Expressed as the reproducibility standard deviation (SD_R); or % reproducibility relative standard
69 deviation (% RSD_R).

70
71 **Recovery**
72 The fraction or percentage of spiked analyte that is recovered when the test sample is analyzed
73 using the entire method.

74
75 **5. Method Performance Requirements:**

76 See table 1.

77
78
79 **6. System suitability tests and/or analytical quality control:**

80 Suitable methods will include blank check samples, and check standards at the lowest point and
81 midrange point of the analytical range.

82
83 **7. Reference Material(s):**

84 Curcumin USP Reference Standard (cat no.: 1151855)
85 Demethoxy-curcumin USP Reference Standard (cat no.: 1173100)
86 Bis-demethoxy-curcumin USP Reference Standard (cat no.: 1075305)
87 Curcuminoids USP Reference Standard (cat no.: 1151866)
88 NIST SRM 3299 Curcuma longa L. (Turmeric) Rhizome
89 NIST SRM 3300 Curcuma longa L. (Turmeric) Rhizome Extract

90
91 Refer to Annex F: *Development and Use of In-House Reference Materials* in Appendix F: Guidelines
92 for Standard Method Performance Requirements, 19th Edition of the AOAC INTERNATIONAL Official
93 Methods of Analysis (2012). Available at: http://www.eoma.aoac.org/app_f.pdf

94
95 **8. Validation Guidance:**

96 For methods based on UV, all compounds in Table 2 must be evaluated for interference.

97 [Appendix D](http://www.eoma.aoac.org/app_d.pdf): Guidelines for Collaborative Study Procedures To Validate Characteristics of a Method
98 of Analysis; 19th Edition of the AOAC INTERNATIONAL Official Methods of Analysis (2012). Available
99 at: http://www.eoma.aoac.org/app_d.pdf

100
101 [Appendix F](http://www.eoma.aoac.org/app_f.pdf): Guidelines for Standard Method Performance Requirements; 19th Edition of the AOAC
102 INTERNATIONAL Official Methods of Analysis (2012). Available at:
103 http://www.eoma.aoac.org/app_f.pdf

104
105 [Appendix K](http://www.eoma.aoac.org/app_k.pdf): Guidelines for Dietary Supplements and Botanicals; 19th Edition of the AOAC
106 INTERNATIONAL Official Methods of Analysis (2012). Available on line at:
107 http://www.eoma.aoac.org/app_k.pdf

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110 **9. Maximum Time-To-Result:** None

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112
113

Table 1: Method performance requirements.

Parameter	Requirement	
Limit of Quantitation (LOQ) (%)	≤ 0.1	
Recovery (%)	97 – 103	
Analytical Range (%)	$\leq 0.1 - 50$	> 50
% RSD _r	≤ 4	≤ 2
% RSD _R	≤ 6	≤ 3

Table 2: Curcuminoids in the presence of other dietary ingredients, for example:

Piper nigrum
Zingiber officinale (ginger)
Capsicum annuum (cayenne pepper)
B-carotene
Lutein
Lycopene
Zeaxanthin

Table 3: Matrices

dried plant material
extracts (purified curcuminoids)
tablets
capsules
softgel capsules
powders
tinctures
liquids

Figure 1: Molecular structure of curcumin

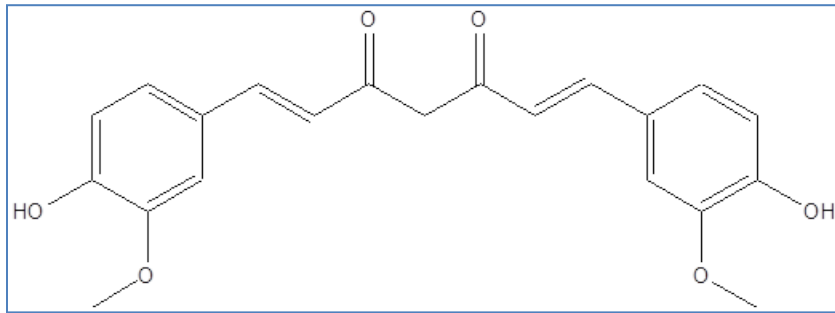


Figure 2: Molecular structure of demethoxycurcumin.

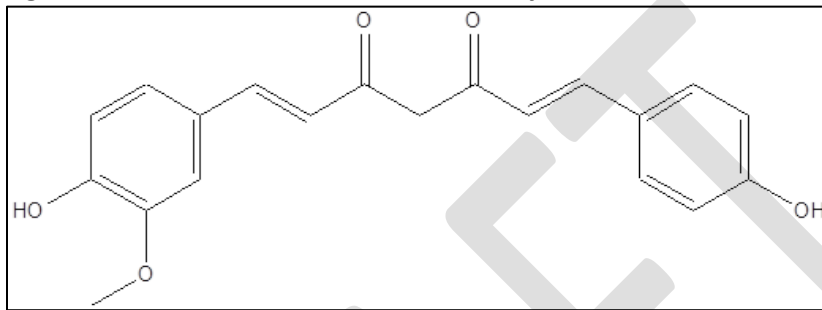


Figure 3: Molecular structure of bisdemethoxycurcumin

